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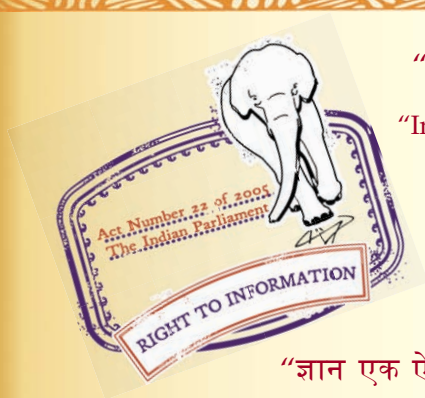
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Indian Standard
METHODS OF TEST FOR
VULCANIZED RUBBERS
PART 7 RESISTANCE TO FLEX-CRACKING
(*First Revision*)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART 7 RESISTANCE TO FLEX-CRACKING

(First Revision)

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Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART 7 RESISTANCE TO FLEX-CRACKING

(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 27 March 1985, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1967 and is now being revised to align it with ISO 132-1975 'Vulcanized rubbers — Determination of resistance to flex cracking (De Mattia type machine)', issued by the International Organization for Standardization (ISO).

0.3 This method of test is intended for use in comparing the resistance of compounds of vulcanized rubbers to flex-cracking when subjected to repeated bending or flexing which causes fatigue failure. Cracks develop in that part of the surface where stresses are set up during flexing, or if that part of the surface initially contains a crack, causes this crack to extend in a direction perpendicular to the stresses.

0.4 The tests prescribed here are intended for use in comparing the resistance of rubbers to the formation and growth of cracks. The relative magnitudes of the two resistances — resistance to crack initiation and resistance to crack growth — differ in different rubbers. It is, therefore, imperative that both the resistance to crack initiation and the resistance to crack growth are measured. A method for determining the resistance to crack growth is prescribed in IS : 3400 (Part 8)-1983*.

0.5 The mean resistance to flex-cracking of a single test piece, with standard conditions of test, should be known with an accuracy of ± 7 percent of the number of flexing cycles (this being \pm twice the percentage standard error of test) but the difference between nominally identical moulded test pieces may be often greater than this.

*Methods of test for vulcanized rubbers: Part 8 Resistance to crack-growth (*first revision*).

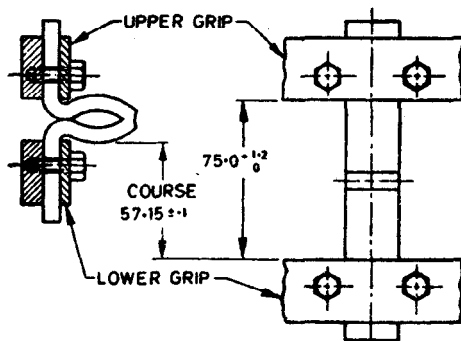
0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2 - 1960*.

1. SCOPE

1.1 This standard prescribes a procedure for comparing the resistance of rubbers to the formation of cracks when subjected to repeated flexing or bending under specified conditions and known periods on the De Mattia type machine.

2. APPARATUS

2.1 The essential features of the De Mattia type machine are given in Fig. 1.



All dimensions in millimetres.

FIG. 1 DE MATTIA TYPE MACHINE

2.2 There shall be stationary parts provided with grips for holding one end of each of the test piece in a fixed position and similar but reciprocating parts for holding the other ends of each of the test pieces. The travel of the reciprocating parts shall be 57.15 ± 0.10 mm and such that the maximum distance between each set of opposing grips is 75.0 ± 1.2 mm.

The reciprocating parts shall be so arranged that their motion is in the direction of and in the same plane as the common centre lines of each opposing pair of grips. The planes of the gripping surfaces of each opposing pair of grips shall remain parallel throughout the motion. The

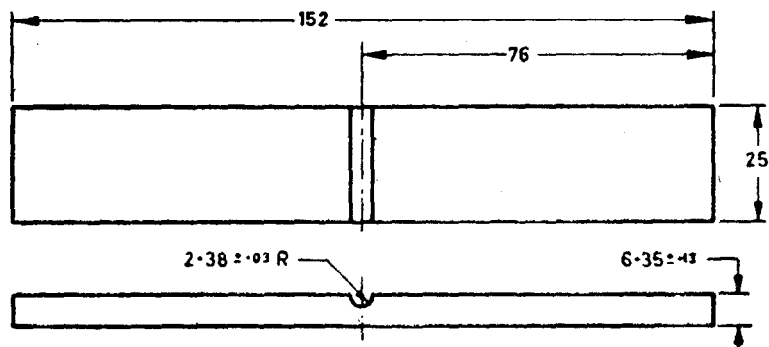
*Rules for rounding off numerical values (revised).

eccentric which actuates the reciprocating parts shall be driven by a constant speed motor to give 300 ± 10 flexing cycles per minute with sufficient power to flex at least 6, but preferably 12, test pieces for one test. The grips shall hold the test piece firmly without undue compression and shall enable individual adjustment to be made to the test pieces to ensure accurate insertion. The test pieces shall be arranged in groups of three or six so that one group is being flexed while the other group is being straightened, thus reducing the vibration in the machine.

2.3 For testing at elevated temperature, the machine may be enclosed in a chamber with temperature controlled to $\pm 2^\circ\text{C}$, if necessary, by using an aircirculator; the temperature to be recorded near the centre of the test piece.

3. TEST PIECE

3.1 The test piece shall be a strip, with a moulded groove as shown in Fig. 2. The strips may be moulded individually in a multiple cavity mould or may be cut from a wide slab having a moulded groove. The moulded groove shall be perpendicular to the grain direction. The groove in the test piece shall have a smooth surface and be free from irregularities from which cracks may start prematurely. The groove shall be moulded into the test piece or slab by a half-round ridge in the centre of the cavity, the ridge having a radius of 2.38 ± 0.03 mm. The results shall be compared only between test pieces having thicknesses agreeing within 0.13 mm when measured close to the groove because the results of the test are dependent upon the thickness of the test piece.



All dimensions in millimetres.

FIG. 2 DE MATTIA TEST PIECE

3.2 At least three (preferably six) test pieces from each rubber shall be tested and the results averaged; one or more test pieces being tested simultaneously with those of other rubber with which the comparison is to be made.

4. TIME LAPSE BETWEEN MANUFACTURE AND TESTING

4.1 For all test purposes, the minimum time between manufacture and testing shall be 16 hours in order to ensure that the material attains dimensional stability due to stress relaxation.

4.2 In order to bind the user and the supplier to a stipulated time for carrying out conformity test for supplied material, the following shall apply.

4.2.1 For non-product test, separate test piece is required for testing. Therefore, the maximum time between manufacture and testing shall be 8 weeks and for evaluation intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

4.2.2 For product test, whenever possible, the time between manufacture and testing should not exceed 4 months. In other cases, tests shall be made within 2 months of the date of receipt of the product by the customer.

4.3 The samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

4.4 Conditioning — For tests at standard laboratory temperature (*see 5.1*), individually moulded test pieces, after preparation as necessary, shall be conditioned at the test temperature for a minimum of 3 hours immediately before testing. The same temperature shall be used throughout, any of the tests or series of tests intended to be comparable. Slab samples shall be similarly conditioned before the test pieces are cut. These test pieces may be either tested immediately or kept at the test temperature until tested.

For tests at elevated temperatures after the conditioning period specified above, the test pieces shall be brought to the test temperature by keeping in a chamber at this temperature for 3 hours and then tested immediately.

5. TEMPERATURE OF TEST

5.1 Tests are normally performed at standard laboratory temperature, namely, $27 \pm 2^{\circ}\text{C}$; although elevated temperatures may often be used with advantage. In the latter case, the test temperature shall be one of the preferred temperatures, such as 40, 50, 70, 85, 100, 125 or 150°C .

6. PROCEDURE

6.1 Separate the pairs of grips to their maximum extent and insert the test pieces so that they are flat and not under tension, with the groove in any particular test piece midway between the two grips in which the test piece is held, and on the outside of the angle made by the test piece when it is bent.

6.2 Start the machine and continue the test with frequent inspection until the first sign of cracking is detected. Record the time or the number of flexing cycles. Carry out this inspection with the grips separated to a distance of 65 mm. Restart the machine and stop after suitable intervals in which the number of flexing cycles is increased by geometric progression, a suitable ratio being 50 percent on each occasion.

6.3 It is not desirable to run the test piece until complete rupture occurs, the preferred method being to grade the severity of cracking by comparison with a standard scale of cracked test pieces as described in 7.

6.4 The test shall not be made in a room which contains any apparatus that generates ozone, or which for any other reason has an ozone content above that in normal indoor air. The motor used to drive the test machine shall be of a type that does not generate ozone.

7. EXPRESSION OF RESULTS

7.1 The comparison with a standard scale of cracked test pieces shall include an assessment of the length, depth and number of cracks and the results shall be recorded as (a) the grade of cracking reached by each test piece on each occasion the machine is stopped, and (b) the number of flexing cycles which have been run, calculated if necessary, from the total period of running. Sometimes, the cracks start developing at the edge of the groove in which case the results should be ignored.

7.2 A photograph of a series of cracked test pieces (see Fig. 3) indicated as Grades A to K and a description of the effects as a guide for the grades which may help in interpreting the photographs, is provided below. Any bloom on the sample shall be reported:

- a) *Grade A* — A few (less than ten) minute cracks have appeared at scattered points on the surface. A lens is not necessary for examining them but the unaided eye is unable to detect that they have any depth. They shall not be confused with mould-marks or specks of dust on the rubber; the latter shall be removed before grading by wiping the test piece with a moistened finger.
- b) *Grade B* — The number of cracks has increased but they still appear to have no depth; they tend to concentrate along the centre-line of the groove and extend to nearly the full width of the test piece.

- c) *Grade C* — The cracks begin to show some depth and their breadth is equal to their length. This grade is regarded as the standard amount of deterioration to which the final result is calculated.
- d) *Grade D* — The cracks have now become so concentrated along the centre-line that a few have coalesced.
- e) *Grade E* — Many of the cracks have now coalesced to form about a dozen cracks, 1 to 2 mm long with a length/breadth ratio of about 2 to 3. This is the most severe degree of cracking which is regarded as satisfactory for grading purposes.

Although the grading *F* to *K* is much more arbitrary, a brief description follows:

- f) *Grade F* — Several cracks have coalesced to form one large crack which releases the surface in the centre of the test piece, thus distorting the top and bottom edges of the groove.
- g) *Grade G* — The large crack has torn its way nearer to the ends of the groove.
- h) *Grade H* — The crack has grown nearer to the ends and has absorbed a number of small ones, thus making its outline indistinct.
- j) *Grade J* — The crack has torn nearly to the ends of the groove.
- k) *Grade K* — The crack has torn right across the groove.

7.3 Logarithmic Method — Reproducible results may be obtained in using the gradings in 7.2 but numerical constants may be associated with the gradings, representing the proportionate increase in time of running to change from one grade to another. If the number of kilocycles for which the machine has run is expressed as logarithm to the base 10, these constants may be added or subtracted to change from one grade to another. Several gradings may be obtained on the same test piece for different periods of running; it is convenient to add the constants corresponding to Grades *A* and *B* and subtract those for *D* and *E*, thus converting all the values to Grade *C*.

These constants for different gradings are:

- a) *Grade A* + 0.35
- b) *Grade B* + 0.15
- c) *Grade C* + 0
- d) *Grade D* - 0.14
- e) *Grade E* - 0.24

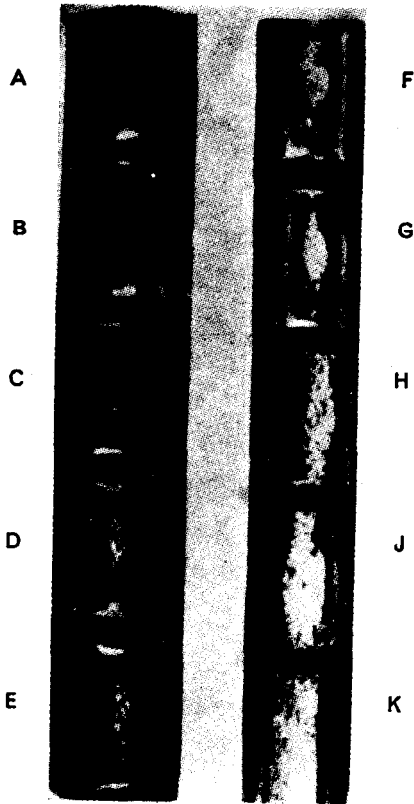


FIG. 3 REFERENCE STANDARD FOR GRADING OF
CRACKED TEST PIECE

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The number of kilocycles at different stages of cracking for a particular test piece may be used to obtain the mean flex-cracking resistance as illustrated in Table 1.

TABLE 1 ILLUSTRATIVE EXAMPLE TO ARRIVE AT MEAN FLEX-CRACKING RESISTANCE

NUMBER OF FLEXING CYCLES	LOG NUMBER OF KILOCYCLES	GRADING	CONSTANT	FLEX-CRACKING RESISTANCE
(1)	(2)	(3)	(4)	(5)
13 500	1.13	A	+0.35	1.48
22 500	1.35	A	+0.35	1.70
31 500	1.50	C	0	1.50
40 500	1.61	C	0	1.61
49 500	1.70	D	-0.14	1.56
58 500	1.77	D	-0.14	1.63
67 500	1.83	D	-0.14	1.69
85 500	1.93	E	-0.24	1.69
Mean value				= 1.61
Standard error of mean value				= 0.03

7.4 The mean flex-cracking resistance represents the logarithm of the mean number of kilocycles required to produce Grade C cracking, the eight individual values lying between 1.48 and 1.70, that is between 30 000 and 50 000 cycles.

7.5 Graphical Method — If no assumption is made as to the manner in which the cracking progresses, the results may be treated graphically, the experimental results being expressed in kilocycles or the logarithm of the observed results. Then results are to be averaged, the arithmetic mean shall be taken, it being noted that the arithmetic mean of the logarithm of the experimental results is equivalent to the geometric mean of the experimental results. It is assumed in the following treatment that either the observed experimental results are being used or that all the experimental results have been converted into their corresponding logarithms, the logarithms then being treated as 'results'. The method is illustrated in Fig. 4.

7.5.1 Considering the individual results for each test piece obtained in the experiment, calculate the average result corresponding to Grades A, B, C, D and E (call these A, B, C, D and E respectively).

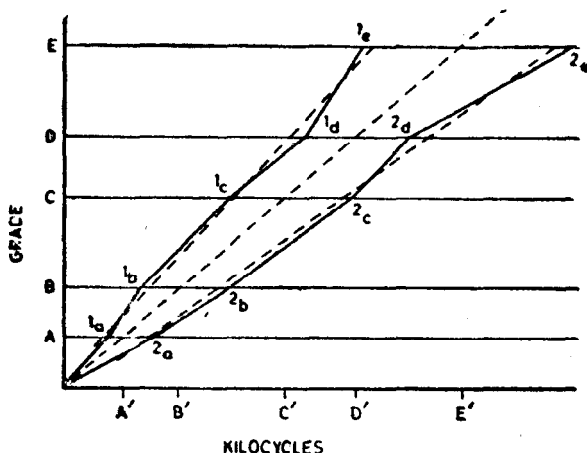


FIG. 4 GRAPHICAL METHOD

7.5.2 Place a linear scale on the abscissa, marked suitably in kilocycles (or logarithms of kilocycles).

7.5.3 Mark five points on the ordinate at distances from the origin proportional to A' , B' , C' , D' and E' respectively, the points being identified by the letters A , B , C , D and E . Draw five lines through A , B , C , D and E parallel to the abscissa.

This may be conveniently accomplished by:

- marking points A' , B' , C' , D' and E' on the abscissa;
- drawing a straight line inclined to the abscissa at a convenient angle;
- placing a series of lines perpendicular to the abscissa through the five marked points and so as to cross the inclined line; and
- drawing through the five points of intersection on the inclined line, a set of five lines parallel to the abscissa and denoted A , B , C , D and E as appropriate.

7.5.4 Consider the results obtained for each compound separately and average the results for the individual test pieces at each of the five grades of cracking. These may be denoted as 1_a , 1_b , 1_c , 1_d and 1_e for compound '1'; 2_a , 2_b , 2_c , 2_d , and 2_e , for compound '2'; and so on.

7.5.5 Plot the results for each compound on the prepared graph paper. For example, for compound '1', the first point is its origin, the second

point has its ordinate on line *A* and abscissa I_a , the third point has its ordinate on line *B* and abscissa I_b , and so on until the sixth and last point with its ordinate on line *E* and abscissa I_e . Similarly for the other compounds, join the points by straight line to give a set of graphs, one graph for each compound.

7.5.6 Draw the best straight line through the points on each graph, ignoring the origin.

7.5.7 The following information may be obtained from the graph:

- a) *Crack initiation time* — The number of kilocycles at which the straight line graph meets the line *A*.
- b) *Crack propagation time* — The number of kilocycles at which the straight line graph crosses the line *E*, minus the crack initiation time.

7.6 Although the method is intended to be applied to the set of experimental results obtained in each experiment, it may be found in practice that the relative positions of the grade lines are sufficiently reproducible in one laboratory for a set of standard grade lines to be determined for that laboratory.

8. REPORTS

8.1 The report shall state:

- a) The average time or the number of flexing cycles to reach each stage of cracking *A* to *E* given in 7, or the mean flex-cracking resistance, by the logarithmic method determined or the crack initiation and propagation, determined by graphical method;
- b) The number of test pieces used; and
- c) The temperature of test.

(Continued from page 2)

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